

Ollscoil na hÉireann, Gaillimh
National University of Ireland, Galway

Summer Examinations, 1999

Third University B.Sc. Examination in Science
(including denominated degrees)

CH302 - Analytical and Industrial Chemistry

First Paper

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and Internal Examiners

Time allowed: **Three** hours

Answer **five** questions

1. Write a short essay on *Laboratory Accreditation*.
2. Answer (a), (b) and (c)
 - a) Discuss the significance of *asymmetry potential* in pH measurement.
 - b) Heyrovsky received the Nobel Prize (1959) for his 1920s work on polarography. Explain why, when developing polarographic methods of analysis, Heyrovsky chose a dropping mercury working electrode (DME).
 - c) The nitrate concentration in an industrial effluent is determined using a nitrate ion-selective electrode. Standards and samples are diluted 20-fold with $0.1 \text{ mol dm}^{-3} \text{ K}_2\text{SO}_4$ in order to maintain constant ionic strength. Nitrate standards of 0.005 and 0.01 mol dm^{-3} give potential readings of -108.6 and -125.2 mV respectively. The sample give a reading of -119.6 mV . What is the concentration of nitrate in the sample?
3. Describe the principle of atomic spectroscopy.

Describe the role of the following components of an atomic absorption instrument: (a) light source; (b) monochromator; (c) detector/measuring system; (d) nebuliser/flame system.

4. Answer (a), (b), (c) and (d)

- a) Explain the following terms as applied to thin layer chromatography:
development
resolution
efficiency
retention factor
detection
- b) Explain the use of a concentrating zone as an aid to sample loading in thin layer chromatography.
- c) Explain how the technique of multiple development can be successful in thin layer chromatography for the separation of closely running components.
- d) Describe the advances in modern TLC which are responsible for making this traditional technique a highly automated quantitative as well as qualitative tool
5. Describe a chromatographic technique for the separation and quantification of a mixture of inorganic ions. Include in your answer a diagram and pay particular attention to the detection system. Explain how simultaneous analysis of a mixture of anions and cations may be performed

6. A sample of benzoic acid (RMM = 122) was thought to be slightly impure. A purity determination was carried out using differential scanning calorimetry. The results are given in the table.

Temperature/K	Area/cm ²
394.37	3.58
394.46	4.39
394.53	5.02
394.61	6.16
394.65	70.4
394.73	8.78
394.8	11.33
394.89	17.52

Total mass of benzoic acid = 2.40 mg; Total measured peak area = 35.4 cm²

Calibration constant = 9.04 mJ/cm²

It was found that the addition of 0.9 cm² to the total and fractional areas resulted in a linear plot of T against 1/F.

Use the data in the table to obtain:

- (a) the melting point of the pure compound
(b) the percentage purity of the sample.

Relevant equation:
$$T_s = T_o - \frac{1}{F} \frac{X_2 R T_o^2}{\Delta H_f}$$

How would you determine the nature of the impurity?

7. Provide clear answers to **each** of the following:

- a) Outline the origin of x-ray fluorescent spectra and describe the system employed for naming x-ray fluorescent lines.
- b) Explain why, in the determinations of iron in an iron/manganese steel by x-ray fluorescence spectroscopy, the calibration plot is linear whereas in a similar determination of the iron in an iron/chromium steel, the calibration plot is non-linear.
- c) Briefly outline the advantages associated with x-ray fluorescence spectroscopy as an analytical technique.

8. Answer (a), (b) and (c).

- a) Describe the types of detector used in gas chromatography. Explain how each detector functions and mention its uses and limitations.
- b) With reference to gas chromatography write brief notes on each of the following:
 - i) height equivalent of a theoretical plate
 - ii) types of column
 - iii) the role of the flow-rate
- c) For a packed column with 2,000 theoretical plates, calculate the width of a chromatographic peak when the retention time (t_r) is 2.00 min. Taking this width as the average, calculate the maximum number of chromatographic peaks that can be resolved per minute from $t_r = 1.5$ min to $t_r = 2.5$ min without overlap at the peak base.